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CLASSIFICATION CHANGED TO:
BY AUTHORITY OF: *Dec 11/60*
BY: *BA/Screen 3-24-50*

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MonN-451 *3A*
Progress Report

MONSANTO CHEMICAL COMPANY
CLINTON NATIONAL LABORATORY

CRNL
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OPERATING DEPARTMENT REPORT

for

Month ending November 30, 1947

PILE OPERATIONS
SEPARATIONS
RADIOISOTOPES

BY

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Date Received: 12/18/47

Date Issued: 12/19/47

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SUMMARY

1. On three different occasions during the month the pile was shut down to locate and discharge slugs with ruptured jackets. In one case the slugs wedged in the row and the pile was down for nine days. The slugs involved in all three failures had been exposed for more than 1,100 days.
2. The excess pile reactivity is about ninety inhours at the present time.
3. The bearings were charged on the No. 2 fan during the past month .
4. Barium Run #22 was completed and shipped in two parts. A total of 2,300 curies was isolated. Some difficulty was experienced during the metathesis step and faulty operation resulted in a loss of about 800 curies.
5. Cell A, Building 706-D is being decontaminated to allow repairs to the equipment.
6. Two hundred isotope shipments were made during November, 1947, to bring the total to 2,004 since the start of the Isotope Distribution Program in August, 1946.

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A. 100 AREA OPERATION

I. Operating Data:

Total Accumulated KWH-----	2,511,518----	2,607,454----	28,214,770
Average KWH/operating hour-----	3735.60-----	3708.14-----	3724.43
Average KWH/24-hour day-----	3488.22-----	3504.64-----	3520.61
Percent lost time-----	6.6%-----	5.5%-----	5.5%
Approx. excess pile reactivity---90 inhours---	35 inhours---		
Slugs charged-----	1530-----	1272-----	9143
Slugs discharged-----	1536-----	1266-----	9191
Product made-----	91.66gm-----	95.16gm-----	1028.85gm
Product discharged-----	16.83gm-----	21.14gm-----	250.00gm

II. Pile Operations:

On three different occasions during the month ruptured slugs were detected. In each case the slugs had been in the pile in excess of 1,100 days in channels with a maximum temperature range of 150° to 190° C. The slugs from the first two channels were discharged without difficulty. When an attempt was made to unload the slugs from the third row, however, considerable trouble was encountered. Thirteen of the slugs in this channel had ruptured and were wedged so tightly in the graphite hole that all conventional means of discharging them failed. It was finally necessary to remove them one at a time. The pile was shut down for approximately ten days because of this slug failure. More information will be included in next month's report.

Water leaking from a "W" tube in Hole 19 shut down the pile by absorbing all of the excess reactivity. After draining the water from the tube, the pile heat and air flow soon dried up

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the moisture in the graphite and normal operations were resumed. The damaged tube was later replaced.

With the exception of the above difficulties, the pile operation was normal throughout the month. The usual scheduled shutdowns were made for the removal and insertion of samples and other miscellaneous work, as well as the discharge of about 1,440 slugs for the 706-D operation, for shipment to the Argonne National Laboratory, and for experimental work.

The excess pile reactivity at the end of November was about ninety inhours as compared with thirty-five inhours at the end of the previous month. The gain of fifty-five inhours was due to the removal of an experimental Cl^{14} factory from Hole 55 and the withdrawal of an experimental furnace from Hole 12.

III. Fan Operation:

The No. 2 fan was shut down toward the end of the month and decontaminated in order that a new bearing could be placed on the shaft. After removal, the old bearing showed a pit approximately $1/16$ " in diameter and $1/32$ " deep in the raceway. The No. 3 fan has operated without difficulty throughout the month.

IV. Radioisotopes:

The number of research and isotope samples loaded into the pile during the month was 204. The following table is a comparison of the samples charged during the months of October and November, 1947:

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OCTOBERNOVEMBER

	<u>Research</u>	<u>Radioisotopes</u>	<u>Research</u>	<u>Radioisotopes</u>
Stringers 13 and 14	20	87	22	101
Hole 22 (Pneumatic Tube)	48	0	69	0
All other holes	<u>13</u>	<u>5</u>	<u>5</u>	<u>7</u>
TOTAL	77	92	96	108

As of November 30, 1947, there are 300 cans of target material in Stringers 13 and 14.

V. Miscellaneous:

1. Beryllium Nitride Canning

Approximately 300 beryllium-nitride pellets were pressed this month. These pellets will be placed into the aluminum cans, welded shut, and tested before shipment to Hanford. A total of one hundred beryllium-nitride slugs were shipped to Hanford on November 26, 1947, for exposure in the pile to produce C^{14} .

2. Calcium Nitrate Canning

A new die is being obtained and the necessary preparations made for pressing calcium nitrate into pellets so that they may be canned and exposed in the Clinton Pile for the production of C^{14} . This work will start soon after the beryllium-nitride canning operation is completed.

3. Waste Metal - (Cold)

Two hundred and thirty-one Hanford-type slugs were stripped and shipped to the Metal Hydrided Company for reclamation. During the past few months an attempt has been made to dispose of all of the waste metal. Less

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than one half ton remains and this will be used by the Technical Division in testing the hot pilot plant.

4. Storage of Irradiated Slugs from 105 Canal

The vault for the storage of old irradiated slugs was recently completed. As soon as possible, all waste slugs which are now stored in the canal will be moved to this new storage vault.

5. Experimental Facilities

West Core Hole

The Physics Division has continued the shielding experiments at the West Core Hole.

B. 706-D AREA

I. Barium (Ba^{140} - 12.5d)

Barium Run #22 was started on November 9, 1947. After some difficulty, it was shipped in two parts on November 22, 1947, and November 27, 1947, respectively. These two shipments contained a total of 2,300 curies at the last separation time. To date we have had no report from Los Alamos concerning the quality of this material.

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In this run we again experienced some difficulty during the metathesis stage of the operation. In order that the spill may be properly cleaned up and also to investigate the condition of the extraction vessel, (A-9) Cell A is being decontaminated.

The analytical summary of Runa #22 is tabulated below:

	<u>Curies*</u>	<u>Percent</u>
Total Curies Dissolved	3995	100.00%
Cell A Losses	537	13.45%
Cell B Losses	263	6.58%
Total Accounted Losses	800	20.03%
Approximate Quantity Shipped	2300	57.57%
Material Balance		77.60%
Losses Unaccounted For	1695	22.40%

* All figures calculated to L.S.T. of second part of shipment.

The spare A-16 blower was installed and the connections will be made prior to the start of the next run.

The UNH solution which has been stored in wooden barrels since 1943 has been transferred into stainless steel drums. All of this material has been turned over to the local Atomic Energy Commission Office for disposition.

II. Radioisotopes:

1. Iodine (I^{131} - 8d) - (Production from Tellurium)

Twenty-four seventy-five-gram cans of tellurium were processed this month to yield approximately 2,100 milli-curies of iodine. Two tellurium cans were processed in each run made this month. Six more seventy-five gram cans of tellurium were in process at the end of the month.

Iodine Development Work - (Fission Product Method)

Four dissolving runs with dead metal have been made in the tantalum-lined dissolvers. Inactive iodine was added to study the behavior of iodine during dissolving. Chemical analyses were not sufficiently precise to get good results on the distribution of iodine, but it was shown that a large amount of the iodine remains in the dissolver, probably being oxidized to iodate. A formic acid reduction followed by an air-steam distillation will probably be necessary to remove most of the iodine. Another run has been made using radioactive iodine; results are not yet available. The metal-dissolving runs were uneventful and the equipment may now be considered ready to dissolve slugs for the fission product columns.

Work is progressing satisfactorily on the installation of the hot hood in 706-C for the final precipitation of iodine. Most of the lead work is completed. The equipment should be ready for experimental work by the latter part of January, 1948.

2. Phosphorus (P^{32} - 14.3d) - (Production from Sulfur)

Twelve cans of irradiated sulfur were extracted this month to produce approximately 2,700 millicuries for November shipments and an excess to meet requirements for the first two weeks of December.

The equipment will be down for approximately the first ten days of December for decontamination and maintenance.

The hood installed sometime ago did not eliminate entirely the high air count which has always been present when this equipment is operated. A new type hood will be installed during the shutdown.

A can of $P_{2}O_{5}$ which had been irradiated in a side-hole tube at Hanford was received and processed. A total of 1,169 millicuries of P^{32} was obtained having a specific activity of 0.79 mc P^{32} /mg P.

Phosphorus Development Work

Work was started on the acetic acid method for extraction of P^{32} from irradiated sulfur. One test has been made on sulfur waste which contains about 0.05 mc P^{32} /g Sulfur. Using air agitation, only two percent of the P^{32} was extracted by the acetic acid. Further tests will be made using mechanical agitation and a fresh sample of irradiated sulfur instead waste material. It is possible that the low concentration of P^{32} in the sulfur waste is non-extractable.

3. Carbon (C^{14} - 5100y) - (From $Ca(NO_{3})_{2}$)

No C^{14} was produced this month as a sufficient stock of this material is on hand. However, it is planned to resume processing when the possibility of contamination from the P^{32} equipment has been eliminated.

C14 Development Work

Data were supplied to the Research Engineering Group so that the project request for alterations to the 204 Annex could be completed and submitted for approval. The project should be ready about December 5, 1947. Little actual experimental work will be done on this process until the equipment is installed so that beryllium and C¹⁴ can be handled safely.

4. Sulfur (S³⁵ - 87.13)

One Na₂S³⁵ production run was made this month. Complete analytical data have not yet been obtained on this run.

5. Fission Products

The all-column fission product equipment has been revised and all work within the cell is complete. The ion chambers, their shields, and accessory equipment will be installed early in December. Several tracer runs will then be made to check the equipment followed by a hot run.

One experimental run has been made on a Zr-Cb fission product fraction using solvent extraction methods in an attempt to separate Zr⁹⁵, Cb⁹⁵, and some unknown activities. Zr⁹⁵ and Cb⁹⁵ apparently can be separated from most of the unknown activities; however, some unknown activities remain with the Cb⁹⁵ in the subsequent Zr-Cb separation.

A Y^{91} fission product column fraction which was contaminated with Cs^{137} , Ru, and 61 has been purified with respect to Cs^{137} by passing it through a resin ion-exchange column. An attempt will be made to remove Pu by oxidation of the Pu followed by extraction into TTA-benzene or removal on an ion-exchange column.

6. Calcium (Ca^{45} - 180d)

No carrier-free Ca^{45} was produced during this period; however, two samples of scandium were purified, shielded with cadmium, and placed in the pile.

A can containing calcium carbonate which has been irradiated at Hanford was received and is now being processed. It is hoped that this material will supply some of the present demand for high specific activity Ca^{45} . Analyses have not yet been obtained on this calcium carbonate.

7. Strontium ($Sr^{89,90}$ - 55d, 30y)

The alcohol-ether HCL wastes from Barium Run 16 was processed this month. The processing equipment was then decontaminated and stored for future use.

8. Iron ($Fe^{55,59}$ - 4y, 44d)

Results have been obtained from M.I.T. on the analysis of the last iron sample which was irradiated 176 days at Hanford. The sample contained a large amount of Fe^{55} because of its long irradiation: 13.8 mc Fe^{55} /g Iron and 2.35 mc Fe^{59} /g Iron, as of November 12, 1947. This

gives a value to the ratio $\text{Fe}^{55}/\text{Fe}^{59}$ of 5.87; the ratio for the previous sample was 2.6. A decision is awaited from the Committee on Medical Usage concerning the maximum allowable dosage of Fe^{55} for human subjects.

Another sample of iron has been received which is smaller than the previous ones and has been irradiated only about sixty days. This should have a more satisfactory $\text{Fe}^{55}/\text{Fe}^{59}$ ratio.

Samples of iron solution are being stored to allow the Fe^{59} to die out, thus providing us with relatively pure Fe^{55} for future sales.

Request have been made to Y-12 for small quantities of iron enriched in Fe^{58} and depleted in Fe^{54} . Bombardment of these samples at Hanford should give us some very high specific activity Fe^{59} .

Work has been discontinued on the process for separating Fe^{59} from cobalt because of the high level radiation encountered and the low yield of this (n, p) reaction in the pile.

9. UX₁ (Th²³⁴ - 24.5d)

Thirty-four millicuries of carrier-free UX₁ were produced from K-25 residue. A large yield loss was suffered from decay during time spent in attempting to improve the chemical purity. Another run is now under way in which F. Bruce's co-precipitation method for pre-purification will be tried.

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10. Zinc (Zn⁶⁵ - 250d)

A cyclotron target backing which has been irradiated with deuterons was machined to remove most of the active metal which was then put into solution. Because of the solder present (to hold the target on) separations for tin and lead must be made as well as the electrolytic separation of copper. The material is now being put through the tin separation step prior to electrolysis for removal of copper.

A second copper cyclotron target backing was recently received and will be processed.

11. Organic Synthesis

Several shipments of labeled methanol have been made during the past month from the stock that was previously built up by the Chemistry Division.

12. Radium-Beryllium Sources

One 0.3-milligram Ra-Be source was prepared during the month.

III. Tank Farm and Burial Ground:

1. Tank Farm

(a) During the past few years acid waste solutions have been run into W-11, the gunite diversion tank. The acid has caused a deterioration of the gunite. A stainless steel tank from the storage area is being used as a replacement.

Recent tests of the atmosphere above the waste solution in the W-11 tank indicates that explosive mixtures are occasionally obtained from solvents run down the hot drains. This problem is being studied and some method will be employed to eliminate this hazard:

- (b) The following listings indicate the movement of solutions in the Tank Farm for the month of November, 1947.

WATER WASTES

<u>Tanks</u>	<u>Capacity</u>	<u>Est. Amt. Rec'd-Nov.</u>	<u>Disposed Of</u>	<u>Discharged To</u>	<u>Free Space</u>
W-1 & 2	8,800 gal.	143,900 gal.	143,900 gal.	Settling Basin & W-5	8,800 gal.

Approximately thirty percent of this waste water was discharged to W-5 tank and the remainder to the Settling Basin. About seventy percent of the water waste came from the Cell 5 operation and the remainder from the 115 Building fan seals.

CHEMICAL WASTES

<u>Tanks</u>	<u>Capacity</u>	<u>Est. Amt. Rec'd-Nov.</u>	<u>Disposed Of</u>	<u>Discharged To</u>	<u>Free Space</u>
W-5-6-8	510,000 gal.	112,400 gal.	207,500 gal.	Settling Basin	231,000 gal.

Approximately eighty percent of this waste was discharged from the 706-D operation, while the balance was from the various hot drains in the other buildings.

METAL WASTES

<u>Tanks</u>	<u>Capacity</u>	<u>Est. Amt. Rec'd-Nov.</u>	<u>Disposed Of</u>	<u>Discharged To</u>	<u>Free Space</u>
W-3-4-7	584,400 gal.	6,400 gal.	0	- - -	62,000 gal.

Approximately 6,300 gallons of metal waste was added to the metal waste tank during the month from the 706-D operation, and approximately one hundred gallons of UNH solution were received from the hot pilot plant at the 205 Building.

SETTLING BASIN

<u>Total Est. Discharge</u>	<u>Total Curies Discharged</u>	<u>Counts/min/ml</u>			<u>Discharged To</u>
		<u>Average</u>	<u>High</u>	<u>Low</u>	
27,338,000 gal.	59	172	1033	10	White Oak Creek

RETENTION POND

275,000 gal.	0.03	9	37	1	White Oak Creek
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2. Burial Ground

(a) Special Burials

- (1) Three shipments of alpha contaminated trash from Dayton.
- (2) One shipment of alpha contaminated trash from Chicago.
- (3) One hundred empty UNH whiskey barrels from the Tank Farm.
- (4) All contaminated material approved by A.E.C. at the East Burial Ground was removed to the West Burial Ground for storage and burial.

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(5) Several bottles of alpha contaminated solution
from 706-A.

(b) Routine Burials

A total of 359 red cans of contaminated trash
as well as the usual miscellaneous materials were
buried during the month.

C. RADIOISOTOPE PRODUCTION AND SHIPMENTS

I. General:

The following table indicates the number of isotope ship-
ments for October and November and a total-to-date figure
since August 1946, the start of the Isotope Distribution
Program.

	OCTOBER 1947	NOVEMBER 1947	TOTAL AUGUST, 1946, to NOVEMBER, 1947, Inc.
Separated Material 706-D Area	155	149	1,387
Irradiation Units 100 Area	64	51	617
TOTAL	219	200	2,004

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The two hundred shipments for November is a decrease of about ten percent from last month which was the highest month to date. The shipments during the past month included eight to the Australian Counsel General, San Francisco, California, for trans-shipment to Australia.

Containers:

A concrete shipping container has been developed which can be fabricated cheaply enough that it will not be necessary for the customer to return it. At the present time this concrete shield will be used only for beta and low energy gamma emitting materials. Further attempts will be made to increase the density sufficiently to allow the shipment of higher energy gamma emitting isotopes.